



# PATENT SPECIFICATION

## 572,140

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### COMPLETE SPECIFICATION

#### Process for the Manufacture of Artificial Silk Resembling Wool

We, **HEBERLEIN & Co., A.G.**, a Swiss Company, of Wattwil, in the Canton of St. Gallen, Switzerland, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

According to a known process, artificial silk resembling wool is made by taking twisted or untwisted artificial silk filaments, winding them with a degree of twist greater than the normal on bobbins, damping them in the highly twisted condition at a raised temperature and then reversing the twist to beyond the zero point. After being allowed to remain in a relaxed condition or when steamed in that condition the artificial silk acquires a permanent, highly crimped "set", resembling that of wool.

It has now transpired that the quality and durability of the resulting products can be further improved and other valuable properties imparted thereto by subjecting highly twisted artificial silk filaments composed of regenerated cellulose to a treatment with powerful swelling agents and then reversing the direction of twist to beyond the zero point preferably after washing (with or without intermediate drying and at a raised temperature, if desired).

Another advantage of the treatment according to the invention is that certain other treatments, such as dyeing or matting, are simplified; whilst at the same time—especially as regards dyeing—the colouring is more evenly distributed and deeper with the result that a saving of dyestuffs is obtained. The improvement in quality resulting from the treatment in accordance with the invention is revealed, inter alia, by the increased elastic ductility of the crimped yarns. The increased persistence of the crimped condition can be convincingly demonstrated by applying the so-called "crimp retentivity" test.

The original length,  $a$ , of the crimped yarn is ascertained by measuring the yarn in a suspended condition. The yarn is then loaded with 0.03 grm. per denier and is dipped for  $\frac{1}{2}$  minute in a tall glass

beaker containing water at 60° C., after which it is again suspended and the length,  $b$ , of the loaded yarn measured. The yarn is then dried in a loose condition and the length  $c$  of the dried yarn is measured in the same manner as for the length  $a$ . The crimp retentivity is

calculated from  $\frac{b-c}{b-a} \cdot 100 = \%$ . The

best way is to take the average of about 10 tests.

It is found that the crimp retentivity is very substantial by the treatment in accordance with the invention. This is especially manifested by an improvement in the resistance of the crimpiness to washing. A further advantage of the process consists in that owing to the treatment of the highly twisted yarn with swelling agents, it shows a considerably lesser tendency to "tangle"—that is, to form intertwining loops—when in the loose state. This is especially beneficial when the highly twisted yarn passes, for further treatment, in the form of hanks or continuous single threads.

Parent materials coming under consideration are: artificial silk filaments consisting of regenerated cellulose—such as viscose or cuprammonium artificial silk—in an untwisted or pre-twisted condition, matted or unmatted, and also pre-dyed, if desired. Whereas, previously, the subsequent dyeing—in the manufacture of such artificial silks resembling wool—gave rise to difficulties, owing to the uneven distribution of the colouring, this is not to be feared in the case of the new process, so that undyed parent materials which are afterwards dyed as required can be used without causing any difficulty.

The untwisted or pre-twisted artificial silk filaments are highly twisted to a greater or lesser degree according to the "count" of the yarn—in practice to about four times the normal twist, or even higher. When the object is to swell the high-twist yarn on bobbins, the operation is preferably performed on perforated bobbins, made of material such as metal, or synthetic resins, which is insensitive to

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the action of the swelling agent used.

As swelling agents, the known customary powerful swelling agents are used, for example: caustic potash lye, caustic soda lye, zinc chloride, and calcium thiocyanate. Sulphuric acid may also be used provided the bobbins—if the treatment is to be carried out on bobbins—are made of acid-proof material. According to whether the swelling agent is to have merely a swelling effect or also a parch-mentising effect, the "feel" and appearance of the yarns are affected. At the same time, it can be noticed that even with the addition of wetting agents or mercerising assistants, provided alkaline lyes are used, the action of the swelling agent on the high-twist yarn proceeds more slowly and less intensively than in the case of normal yarns. Consequently, swelling agents may be used in such concentration as would, otherwise, be harmful to artificial silks—for example, caustic soda lye of 10—18° Bé strength. Caustic potash lye (30° Bé) and caustic soda lye (30° Bé) have been found to be particularly effective swelling agents, though other concentrations, for example from 6° Bé to over 30° Bé, can also be used. Solutions of zinc chloride or calcium thiocyanate (60—70% strength) are also suitable, likewise weaker concentrations when, for example, the treatment is conducted at higher temperatures. In the case of caustic soda lye and caustic potash lye, a treatment temperature of about 80° C. has been found suitable, though good results are obtainable by operating at room temperature. The duration of the treatment is, in general, a few minutes, for example 3 minutes, at 80° C., in the case of 30° Bé caustic potash.

The treatment with swelling agents can be advantageously carried out on the bobbins on which the high-twist has been imparted, a typical method being to draw or force the swelling agent through the perforated bobbins. The high-twist yarn may, however, be reeled and treated in hank form under tension. The yarn may also be drawn in a continuous length over guide rollers, through a bath of the swelling agent.

After the swelling treatment the swelling agent is preferably washed out and the yarn dried. This can be done on the bobbins, if the swelling treatment has been carried out thereon. However, according to the nature of the swelling agent the washing may be omitted and deferred until after the drying. The drying is advantageously conducted at a raised temperature. After being washed and dried the yarn is twisted in the reverse direction to beyond the zero point, mostly

to the extent of about 70—80 additional turns, whereupon the goods can, for example be reeled into hank form. If desired, the reverse twist can also be applied without previous drying. If improving treatments—for example dyeing or matting—are contemplated, they can be advantageously applied after the swelling treatment, between washing and drying, and on the bobbins if desired.

After the reversed twist beyond the zero point, the yarns may also be cut up into staple fibres, and spun and further treated, in that form. In order to reduce their capacity for swelling, they may be treated, after being twisted in the reverse direction, with formaldehyde at raised temperatures and in the presence of acid catalysts, in known manner.

The following Examples illustrate the invention without however exhausting its possible applications.

#### EXAMPLES:

1. Viscose yarn (titre 450 deniers) is twisted 1500 turns per metre, in the S-direction, and wound direct on to perforated bobbins of alkali-proof materials. The bobbins charged with the twisted yarn are then slipped on to a suitable frame and placed into a dyeing apparatus, such as the Ohermayer or Franklin apparatus. Caustic potash lye, of the density 30° Bé (at 15° C.), and preheated to 80° C., is then caused to circulate through the bobbins under pressure, 1—1 atmos. being usually sufficient. After the treatment has continued for 4 minutes, the lye is discharged from the apparatus, and the yarn material is freed from alkali by means of hot water. Neutralisation is completed by means of acetic acid, the material is thoroughly washed with cold water, the surplus liquid is removed by aspiration, and, finally, the material is dried by forcing warm air through the bobbins. The yarn is next unwound, and twisted, in the Z-direction, to the extent of 70—80 turns per metre, beyond the zero point, being then reeled, and steamed, in hank form, for 5—10 minutes, without pressure. A strongly crimped, bulky yarn, of good wool character, is obtained.

2. Artificial silk yarn, twisted as in Example 1, is treated with lye and given a reversed twist. After being reeled, it is treated, in hank form, for 15 minutes at 30—40° C., in a bath containing 3 grms. of oil soap per litre, being then well washed, centrifuged, and dried in a freely suspended condition. A strongly crimped yarn, of wool character, is obtained, which does not undergo any substantial alteration, even after repeated washings.

3. Artificial silk yarn, with a titre of 300 deniers, and pre-dyed with vat dyes, is twisted 1800 turns per metre, in the S-direction, and is introduced, in the form of cross-reel on perforated bobbins of lye-proof material into an Obermayer dyeing apparatus. Caustic potash lye (16° Bé), containing, per litre, 10 cc. of Flerhenol merceriser (mercerising assistant, made by the Fleschwerke Aktiengesellschaft, Frankfurt a/M., and consisting of a mixture of crude cresol and fat solvents) is caused to circulate through the apparatus for 10 minutes at room temperature, the surplus lye being then discharged from the vessel, and the yarn freed from alkali by washing with water at about 60—70° C. It is then neutralised with dilute hydrochloric acid, and washed until neutral. After drying, a twist in the Z-direction of 80 turns per metre, beyond the zero point, is imparted to the yarn. Two such yarns are then twisted, with a twist of 50 turns per metre in the S-direction, and entered, in hank form, into a bath containing, per litre, 1 gm. of the substance known under the Registered Trade Mark "Igepon T" (a sodium salt of the methyl-tauride of higher fatty acids), worked therein for 10 minutes at 50° C., rinsed, centrifuged, and dried in an unstretched condition. In this manner, a coloured, voluminous and soft twist is obtained, the fibrils of which exhibit strong crimping. The effect offers good resistance to washing.

4. Viscose yarn (titre 400 deniers) is twisted 1600 turns per metre, in the S-direction and wound direct on to perforated conical bobbins. These shells are placed on a suitable rack, as described in Example 1, and introduced into a dyeing apparatus, wherein they are treated with caustic soda lye (23.5% strength) for 3 minutes at 15° C. To improve the wetting effect, 10 cc. of Flerhenol merceriser or some other mercerising assistant such as the phenolic product known under the Registered Trade Mark "Mercerol" is added for each litre of the lye. After the surplus lye has been run off, the yarn is thoroughly rinsed with hot water, and then dyed, in the following manner, with substantive dyestuffs, such as Direct Sky Blue.

A bath is prepared, containing 1 gm. of "Igepon T" per litre, and dyeing is conducted at boiling heat, the previously dissolved dyestuff being slowly added to the dyebath in different portions. When the desired depth of shade and sufficient penetration have been attained, the yarn is given a good rinsing with cold water, hydrogenated, and dried by warm air. The

yarn is then unwound, and twisted to 60 turns per metre, in the Z-direction, beyond the zero point, and reeled, the hanks being lightly damped. The resulting yarn is very soft, voluminous, and the individual fibrils are strongly crimped. Light washing, such as is usual for direct-dyed textiles, has practically no effect on the crimping effect.

5. Viscose yarn (titre 450 deniers) is given a twist of 1400 turns per metre, in the S-direction, and, in this highly-twisted condition, is passed, as individual filaments, through a bath of caustic potash lye (with a density of 16° Bé at 15° C.), at a temperature of 50° C., the yarn being then squeezed, dried in a current of air at 60—70° C., and reeled. The hanks are then washed with hot water, neutralised with dilute acid, to neutralise traces of alkali, well rinsed and dried. The washing and neutralisation are advantageously performed with the hanks under slight tension. The yarn is next given a reverse twist of 100 turns per metre, in the Z-direction, beyond the zero point, and made into a twist, of 70 turns per metre, in the S-direction, with a 100 den. viscose crêpe yarn. This new twist is very suitable for the manufacture of novel fabrics, since it can be used as weft in a correspondingly open warp, or as both warp and weft yarns. The fabrics so obtained are creped in a soap bath, after-bleached if required, dyed, and dried under light tension. The resulting fabrics are very elastic, bulky and of woolly character.

6. Cuprammonium silk (titre 360 deniers) is twisted 1500 turns per metre in the S-direction and then led in the form of a single thread, through a bath of caustic potash lye, with a density of 14° Bé at 15° C. The lye is heated to 70° C., and 10 cc. of Flerhenol merceriser per litre added, in order to improve the wetting capacity. The surplus lye is squeezed out and, in order to lengthen the period of action of the lye, the yarn is advantageously carried over a reel in the lye-removing bath. The time during which the lye is in action should be at least  $\frac{1}{2}$ —1 minute. The lye is removed with hot water, a short neutralising being then given with dilute acetic acid, followed by a rinsing with cold water. The yarn is then squeezed, dried in a warm current of air, or by being passed over hot cylinders, and wound on to bobbins, followed by reeling into hanks with a twist of up to 70 turns per metre, in the Z-direction. The hanks are soaped, without tension, for 10 minutes at 50° C., rinsed, centrifuged, and dried in a state of free suspension, a bulky yarn, of woolly character, being obtained. The strong

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crimping of the individual fibrils deflects the light in a diffusive manner, and the modified textile material has then only a slightly discernible sheen.

- 5 7. The cuprammonium silk yarn, with a high twist imparted by the method described in Example 6, and treated with 16° Bé caustic potash lye, is washed and dried, given a reverse twist of 40 turns per metre in the Z-direction, reeled, steamed, without pressure, for 10 minutes in a freely suspended condition and then formed into a twist yarn with a 120 den. artificial silk yarn (of normal twist), by imparting a twist of 40 turns per metre in the Z-direction. In this manner, an interesting mixed twist is obtained, which is very suitable for weaving. Instead of being used as a mixed twist, the treated cuprammonium silk yarn can also be mixed with any ordinary yarn, and then woven.

8. Viscose silk (titre 450 deniers) is twisted as described in Example 1, treated on bobbins with hot caustic potash lye, given a reversed twist, reeled and steamed. The yarn, in hank form, is then worked for 10 minutes in a cold bath of the following composition: Concentrated commercial formaldehyde, 100 cc., Aluminium thiocyanate solution (17° Bé strength) 40 cc., and water 860 cc. (1000 cc. in all).

- The surplus liquid is removed by centrifuging, the loosely suspended yarn is dried at 60—70° C., and is then heated at 120° C. for 15 minutes. The product so obtained is distinguished by especially good fastness to washing, and is very similar to wool, both in appearance, heat conductivity and capacity for absorbing water. Owing to the reduced capacity for swelling the hygroscopic properties are substantially lower than those of the parent material.

9. Viscose yarn (450 deniers) is twisted 1500 turns per metre, in the S-direction, wound directly on to perforated bobbins and introduced, thereon, into a dyeing apparatus. A solution of zinc chloride (prepared by dissolving 4 parts by weight of powdered zinc chloride in 6 parts by weight of water) is then forced through the bobbins at temperatures between 90° and 95° C. After acting for 5 minutes, the zinc chloride solution is run out of the vessel. The yarn material is then completely freed from zinc chloride by washing with very dilute acetic acid, followed by a good washing with cold water, and is dried on the bobbins. It is then twisted in reverse, to 80 turns per metre in the Z-direction, beyond the zero point. After being reeled into hanks weighing about 30 grms., the material is steamed, in a loosely suspended condition, without pressure. The yarn so obtained is of

good woolly character, voluminous, soft and highly elastic.

We are aware that Specification No. 442,073 discloses a process for crimping filaments or fibres comprising imparting twist to a travelling yarn containing filaments or fibres, applying a setting medium, which may be a softener for the substance of the yarn, to said travelling yarn over part of its length actually receiving the twist imparted and subsequently twisting the yarn in the direction reverse to that of the twist imparted.

The said yarn may be collected in its set, twisted form and then twisted in the reverse direction to effect any desired amount of untwisting, which, in the case of continuous filament yarns, may even be greater than the amount of twist in the set yarn.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. Process for the manufacture of artificial silk resembling wool, characterised in that highly-twisted artificial silk filaments of regenerated cellulose are subjected to a treatment with powerful swelling agents and afterwards given a reverse twist beyond the zero point.

2. Process according to claim 1, characterised in that the yarns are washed before the reversed twist is imparted.

3. Process according to claim 1, characterised in that the yarns are washed and dried before the reversed twist is imparted.

4. Process according to claims 1 and 2, characterised in that the yarns are dried at a raised temperature.

5. Process according to claim 1, characterised in that the high twist is imparted to the filaments on bobbins made of material insensitive to the swelling agent used.

6. Process according to claim 1, characterised in that the high-twist filaments are reeled and are treated, under tension, in the form of hanks.

7. Process according to claim 1, characterised in that, after the treatment with swelling agents, finishing treatments such as dyeing or matting are given between the operations of washing and drying.

8. Process according to claim 1, characterised in that the treatment with swelling agents is conducted at a raised temperature.

9. Process according to claim 1, characterised in that the yarns are dyed after the treatment with swelling agents.

10. Process according to claim 1, characterised in that after the reversed

twist has been imparted the yarns are subjected to a treatment with formaldehyde, at raised temperatures and in the presence of acid catalysts.

5 11. Process according to claim 1, characterised in that caustic potash lye is used as the swelling agent.

12. Process according to claim 1, characterised in that caustic soda lye is

used as the swelling agent.

13. Artificial fibres when produced by the process as claimed in the preceding claims.

Dated the 29th day of March, 1943.

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888

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